

FORM PTO-1390
(REV. 9-2001)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

Mo6885/LeA 33,410

U.S. APPLICATION NO. (If known, see 37 CFR 1.5

To be Assigned 104030676

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP00/06599

12 July 1999 (12.07.99)

16 July 1998 (16.07.98)

TITLE OF INVENTION

Adjustable Jet Dispenser for Producing Aqueous Two-Component Polyurethane Paint Emulsions

APPLICANT(S) FOR DO/EO/US ; KLINKSIEK, Bernd; SCHLEENSTEIN, Dieter; HOVESTADT, Wieland and VOM FELDE, Michael

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☒ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

Abstract page

Form PTO 1449 with references

Drawings 4 sheets containing Figures 1-5

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)

INTERNATIONAL APPLICATION NO.

ATTORNEY'S DOCKET NUMBER

To be assigned **107030676**

PCT/EP00/06599

Mo6885/LeA 33,410

21. ☒ The following fees are submitted:

CALCULATIONS PTO USE ONLY

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO. \$1040.00

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$890.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 890.00

Surcharge of **\$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$

CLAIMS

NUMBER FILED

NUMBER EXTRA

RATE

\$

Total claims

4 - 20 =

x \$18.00

\$

Independent claims

1 - 3 =

x \$84.00

\$

MULTIPLE DEPENDENT CLAIM(S) (if applicable)

+ \$280.00

\$

TOTAL OF ABOVE CALCULATIONS =

\$ 890.00

☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above
are reduced by 1/2.

\$

SUBTOTAL =

\$

Processing fee of **\$130.00** for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

TOTAL NATIONAL FEE =

\$ 890.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$

40.00

TOTAL FEES ENCLOSED =

\$ 930.00

Amount to be
refunded:

\$

charged:

\$

a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 13-3848 in the amount of \$ \$930.00 to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. 13-3848. A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card
information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR
1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:



00157

PATENT TRADEMARK OFFICE

SIGNATURE

Thomas W. Roy

NAME

29,582

REGISTRATION NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES RECEIVING OFFICE

| | |
|-------------------------------|-------------------|
| Date | 11 January 2002 |
| International Application No. | PCT/EP00/06599 |
| Attorney Docket No. | Mo6885/LeA 33,410 |


I. Certification under 37 CFR 1.10 (if applicable)

JC13 Rec'd PCT/PTO 11 JAN 2002

| |
|-----------------------------|
| ET671474685US |
| Express Mail mailing number |

| |
|-----------------|
| 11 January 2002 |
| Date of Deposit |

I hereby certify that the application/correspondence attached hereto is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to Assistant Commissioner for Patents, Washington, D.C. 20231.

| |
|---|
|  |
| Signature of person mailing correspondence |

| |
|--|
| Donna J. Veatch |
| Typed or printed name of person mailing correspondence |

II. ☒ New International Application

| | |
|-------|--|
| TITLE | Adjustable Jet Dispenser for Producing Aqueous Two-Component Polyurethane Pain Emulsions |
|-------|--|

| |
|--|
| Earliest priority date (Day/Mon/Year) |
| 16, July, 1999 |

SCREENING DISCLOSURE INFORMATION: In order to assist in screening the accompanying international application for purposes of determining whether a license for foreign transmittal should and could be granted and for other purposes, the following information is supplied. (Note: check as many boxes as apply):

- A. ☐ The invention disclosed was not made in the United States.
- B. ☐ There is no prior U.S. application relating to this invention.
- C. ☐ The following prior U.S. application(s) contain subject matter which is related to the invention disclosed in the attached international application. (NOTE: priority to these applications may or may not be claimed on form PCT/RO/101 (Request) and this listing does not constitute a claim for priority.)

| | | | |
|-----------------|--|----------|--|
| application no. | | filed on | |
| application no. | | filed on | |

- D. ☐ The present international application ☐ contains additional subject matter not found in the prior U.S. application(s) identified in paragraph C. above. The additional subject matter is found on pages and ☐ DOES NOT ALTER ☐ MIGHT BE CONSIDERED TO ALTER the general nature of the invention in a manner which would require the U.S. application to have been made available for inspection by the appropriate defense agencies under 35 U.S.C. 181 and 37 CFR 5.1. See 37 CFR 5.15

III. ☐ A Response to an Invitation from the RO/US. The following document(s) is(are) enclosed:

- A. ☐ A Request for An Extension of Time to File a Response
- B. ☐ A Power of Attorney (General or Regular)
- C. ☐ Replacement pages:

| | | | | | |
|-------|--|-----------------------------|-------|--|-----------------|
| pages | | of the request (PCT/RO/101) | pages | | of the figures |
| pages | | of the description | pages | | of the abstract |
| pages | | of the claims | | | |

- D. ☐ Submission of Priority Documents

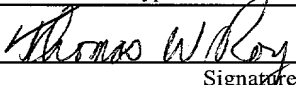
| | | | |
|-------------------|--|-------------------|--|
| Priority document | | Priority document | |
|-------------------|--|-------------------|--|

- E. ☐ Fees as specified on attached Fee Calculation sheet form PCT/RO/101 annex

IV. ☐ A Request for Rectification under PCT 91 ☐ A Petition ☐ A Sequence Listing Diskette

- V. ☒ Other (please specify): Preliminary Amendment w/Abstract page
PTO 1449 with listed references
Drawings 4 sheets containing Figures 1-5

The person
signing this
form is the:

| | |
|---|---|
| <input type="checkbox"/> Applicant | Thomas W. Roy |
| <input checked="" type="checkbox"/> Attorney/Agent (Reg. No.) 29,582 | Typed name of signer |
| <input type="checkbox"/> Common Representative |  Signature |

PATENT APPLICATION
Mo-6885
LeA 33,410

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF)
BERND KLINGSIEK ET AL) PCT/EP00/06599
SERIAL NUMBER: TO BE ASSIGNED)
FILED: HEREWITH)
TITLE: ADJUSTABLE JET DISPERSER FOR)
PRODUCING AQUEOUS TWO-)
COMPONENT POLYURETHANE)
PAINT EMULSIONS)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to the examination of the subject application, kindly amend the
application as follows:

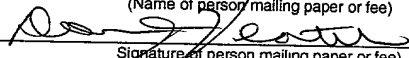
"Express Mail" mailing label number ET671474685US

Date of Deposit January 11, 2002

I hereby certify that this paper or fee is being deposited with the United States
Postal Service "Express Mail Post Office to Addressee" service under 37 CFR
1.10 on the date indicated above and is addressed to the Assistant Commissioner
of Patents and Trademarks, Washington, D.C. 20231

Donna J. Veatch

(Name of person mailing paper or fee)


Signature of person mailing paper or fee)

IN THE SPECIFICATION:

Please delete the current title and replace with the following:

--ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS TWO-
COMPONENT POLYURETHANE PAINT EMULSIONS--.

Please replace the Abstract on page 13 with the following new abstract:

--ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS TWO-
COMPONENT POLYURETHANE PAINT EMULSIONS

ABSTRACT OF THE DISCLOSURE

The invention relates to a process for the production of aqueous two-
component polyurethane lacquer emulsions, with variable throughput, and to the use
thereof as high-quality lacquers and coatings.--

A new abstract page is included herewith.

At page 1, line 3 insert the heading --BACKGROUND OF THE INVENTION--.

At page 4, line 23 insert the heading --SUMMARY OF THE INVENTION--.

At page 5, line 1 insert the heading --DETAILED DESCRIPTION OF THE
INVENTION--.

At the end of page 11, line 12, insert the heading --BRIEF DESCRIPTION OF
THE DRAWINGS-- and the following paragraphs:

--Figure 1 describes one embodiment of the invention and shows a cross-section
through a dispersing device according to the invention, with mixing nozzle connected
upstream.

Figure 2 describes one embodiment of the invention and shows a cross-section
through a variant of the dispersing device in Figure 1 with opposite rows of axially
displaced bores. It also shows a detailed drawing of the nozzle in Figure 2 (lateral view)
in order to illustrate the geometry of the nozzle.

Figure 3 describes one embodiment of the invention and shows a cross-section
through a variant of the dispersing device in Figure 1 with slots 16, 16' and it also shows
a detailed drawing of the nozzle in Figure 3 (lateral view) in order to illustrate the
geometry of the nozzle.

Figure 4 describes one embodiment of the invention and shows the scheme of a coating unit with several dispersing devices according to the invention.

Figure 5 describes one embodiment of the invention and shows a graph representing the average particle size as a function of the homogenizing pressure for various dispersing devices.--

IN THE CLAIMS:

Cancel Claim 1.

Kindly add the following claims:

- 2. A process for the production of an aqueous two-component polyurethane coating emulsion comprising pumping a mixture of at least one polyisocyanate and an aqueous binder dispersion comprising isocyanate-reactive groups under a pressure of from 1 to 30 MPa through an adjustable jet disperser.
3. The process of Claim 2 wherein the jet disperser has a variable throughput.
4. The process of Claim 2 wherein the jet disperser comprises nozzle bores or slots.
5. The process of Claim 4 wherein the bores or slots can be opened and closed.--

REMARKS

Claim 1 has been cancelled and replaced by Claims 2-5. These claims are supported by the original claim.

An early action of the merits is respectfully requested.

Respectfully submitted,

By Thomas W. Roy
Thomas W. Roy
Attorney for Applicants
Reg. No. 29,582

Bayer Corporation
100 Bayer Road
Pittsburgh, Pennsylvania 15205-9741
(412) 777-8345
FACSIMILE PHONE NUMBER:
(412) 777-8363

s:/sr/dp0060

VERSION WITH MARKINGS TO SHOW CHANGES MADE:

IN THE SPECIFICATION:

Please delete the current title and replace with the following:

--ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS TWO-
COMPONENT POLYURETHANE PAINT EMULSIONS--.

Please replace the Abstract on page 13 with the following new abstract:

--ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS TWO-
COMPONENT POLYURETHANE PAINT EMULSIONS

ABSTRACT OF THE DISCLOSURE

The invention relates to a process for the production of aqueous two-
component polyurethane lacquer emulsions, with variable throughput, and to the use
thereof as high-quality lacquers and coatings.--

A new abstract page is included herewith.

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At page 4, line 23 insert the heading --SUMMARY OF THE INVENTION--.

At page 5, line 1 insert the heading --DETAILED DESCRIPTION OF THE
INVENTION--.

At the end of page 11, line 12, insert the heading --BRIEF DESCRIPTION OF
THE DRAWINGS-- and the following paragraphs:

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through a dispersing device according to the invention, with mixing nozzle connected
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through a variant of the dispersing device in Figure 1 with opposite rows of axially
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geometry of the nozzle.

Figure 4 describes one embodiment of the invention and shows the scheme of a coating unit with several dispersing devices according to the invention.

Figure 5 describes one embodiment of the invention and shows a graph representing the average particle size as a function of the homogenizing pressure for various dispersing devices."

IN THE CLAIMS:

Claim 1 has been canceled.

The following new claims have been added:

- 2. A process for the production of an aqueous two-component polyurethane coating emulsion comprising pumping a mixture of at least one polyisocyanate and an aqueous binder dispersion comprising isocyanate-reactive groups under a pressure of from 1 to 30 MPa through an adjustable jet disperser.
3. The process of Claim 2 wherein the jet disperser has a variable throughput.
4. The process of Claim 2 wherein the jet disperser comprises nozzle bores or slots.
5. The process of Claim 4 wherein the bores or slots can be opened and closed.--

ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS
TWO-COMPONENT POLYURETHANE PAING EMULSIONS

ABSTRACT OF THE DISCLOSURE

The invention relates to a process for the production of aqueous two-component polyurethane lacquer emulsions, with variable throughput, and to the use thereof as high-quality lacquers and coatings.

10030675 011102

Adjustable jet disperser for the production of aqueous two-component polyurethane lacquer emulsions

5 The invention relates to a process for the production of aqueous two-component polyurethane lacquer emulsions, with variable throughput, and to the use thereof as high-quality lacquers and coatings.

10 Two-component polyurethane lacquers (2C-PUR lacquers) are not mixed until shortly before application, because of the limited processing time (working life) of the lacquers. Depending on the reactivity of the lacquer systems, the said working life may be from several minutes to several hours.

15 While such two-component systems have in the past been used in solution in organic solvents, a number of water-dispersible two-component systems have recently been developed. Water-dispersible two-component systems normally consist of a resin component containing hydroxyl groups (binder, polyol), and a polyisocyanate component (hardener, crosslinking agent). The hydroxy-functional resin component is generally present in the form of an aqueous dispersion, and the polyisocyanate component is generally in the form of an anhydrous one-hundred-percent component or is in solution in a solvent. Such systems, which can also be used within the scope of the present invention, are disclosed, for example, in EP-A 358 979, 496 205, 469 389, 520 266, 540 985, 542 105, 543 228, 548 669, 562 282 and 583 728. A disadvantage of those lacquer systems is that the known lacquer quality of two-component systems based on purely organic solvents is not achieved in some fields of application. That is true especially of fields of application in which particularly high optical properties and resistance properties are required.

25
30 In order to achieve lacquer surfaces of high quality, it is known to use lacquer dispersions the particle size of which is as small as possible. In aqueous two-component polyurethane lacquers, therefore, polyol dispersions having a sufficiently

small particle size of less than 500 nm, preferably from 10 to 200 nm, are generally used. Dispersion of the isocyanate component, which is hydrophobic *per se*, does not take place until shortly before application of the lacquers, since the polyisocyanate component reacts with water and therefore has only a limited storage stability in the presence of water. Dispersion of the polyisocyanate component, which is hydrophobic *per se*, in the aqueous hydroxy-functional resin dispersion by means of conventional static mixing devices causes considerable difficulties, however. The reason is that the isocyanate component is stabilised during the emulsification at the surface of emulsion particles that have already formed, so that the superficial stabilising layer stands in the way of further division. Aqueous two-component polyurethane lacquer emulsions therefore normally have a bimodal particle size distribution, with a first distribution maximum at a particle size corresponding to the hydroxy-functional resin dispersion and a second distribution maximum at a particle size greater than 10,000 nm (isocyanate component), considerable portions having particle sizes greater than 20,000 nm still being present.

Polyisocyanates that have already been rendered hydrophilic by chemical modification and polyisocyanates containing external emulsifiers have also been developed; although such polyisocyanates permit markedly simpler dispersion to an average particle size of less than 1000 nm using static mixing devices, they yield hardened lacquer films that have resistance properties which are inadequate for many fields of application. Lacquer films having good resistance properties, however, are obtained only by the use of hydrophobic polyisocyanate components.

On the basis of the conception that the dispersibility of the isocyanate component is limited by the stabilisation reaction which takes place at the surface of particles that are already present, possible ways have been sought of achieving as finely divided a distribution as possible within periods of time which are so short that noticeable superficial stabilisation does not yet occur. In particular, warming which accelerates

the reaction of the polyisocyanate component with water is also to be avoided during the dispersion.

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5 In DE-A 19 510 651 it is described that, after pre-mixing of the hydroxy-functional binder dispersion with the polyisocyanate component, water, optionally additional solvent and, optionally, other lacquer auxiliaries known *per se*, the resulting pre-emulsion, of which no particular requirements are to be made, is forced at high pressure through a nozzle having small measurements in at least one dimension. There are mentioned as homogenising nozzles slit-type nozzles, circular-slot nozzles
10 or hole-type nozzles. Special preference is given to the use of a jet disperser according to EP-A-101 007, since very finely divided dispersions are formed even at relatively low pressures. The pressure to be applied is to be from 1 to 30 MPa (from 10 to 300 atmospheres), in the case of the jet disperser preferably from 1 to 8 MPa, especially from 2 to 6 MPa. By means of that emulsification it is possible to produce
15 two-component polyurethane lacquer emulsions which are stable for several hours and which exhibit a considerably improved surface quality after application and hardening.

20 It is also possible to reduce the content of solvent in the dispersion considerably and preferably to dispense with rendering the polyisocyanate component hydrophilic. In particular, dispersions having a solvent content of less than 15 % can readily be produced in accordance with the invention. Depending on the pressure applied during the dispersion, the number of nozzle passages and the two-component system used, it is also possible to produce emulsions that are completely free of solvents and
25 agents imparting hydrophilicity.

The high lacquer surface qualities achievable with the process according to the invention can be related directly to the particle size distribution of the emulsions.

In DE-A 19 510 651, the use of the jet disperser having fixed dimensions is described in Figures 1, 2, 3, 4 and 5. Figure 6 describes a particular form in which the bores are freed or closed off by a movable application pipe. This form has proved to be impractical for use. It has been found that such homogenising nozzles must be adjustable very rapidly in fractions of a second in order to produce the same emulsion quality continuously when the amounts deposited vary.

It has been found that motor vehicle bodies, for example, can be lacquered especially advantageously at a very high quality if the polyisocyanate is continuously emulsified in the aqueous polyol component immediately before being introduced into the spray gun or atomiser bell. Problems arise, however, if the amount of lacquer taken varies within very short intervals of time owing to the geometry of the motor vehicle body.

The object of the invention was, therefore, to make available a mixing apparatus for aqueous two-component polyurethane lacquers having a high lacquer quality, which mixing apparatus continuously produces the same emulsion qualities when the amounts deposited vary.

Surprisingly, it has been possible to achieve that object by means of the embodiment of a jet disperser described in greater detail below.

The invention provides a process for the production of aqueous two-component polyurethane lacquer emulsions, with variable throughput, based on aqueous binder dispersions containing isocyanate-reactive groups and on polyisocyanates, by mixing the two components, characterised in that the mixture is pumped under a pressure of from 1 to 30 MPa through an adjustable jet disperser having nozzle bores or slots which can be connected through or shut off.

According to the invention, a mixing nozzle (1) for the polyisocyanate is advantageously provided immediately upstream of the jet disperser (Figure 1) and the crude emulsion is produced by injection into the polyol component. Immediately downstream there is provided a further screen (2), which ensures that the crude emulsion is of good quality and prevents coarse fractions.

The core element of the apparatus according to the invention is a ceramics sleeve (3) having the homogenising bores (4) and the ceramics piston (5). It has been found that the ceramics components must be ground in to fit very accurately in order to avoid leakage between the piston and the sleeve. It has been found that components made of steel do not permit a tight seal and thus allow individual bores to be connected through. It has also been found that the bores at the inlet side should be ground with very sharp edges. Suitable ceramics materials are metal oxides such as, for example, zirconium oxide or even harder materials. It has also proved advantageous to provide a flushing bore (6), which permits a large mass flow on flushing. Flow through the ceramics sleeve may be either from the inside to the outside or from the outside to the inside. In order to avoid a film of lacquer on the piston when the apparatus is at a standstill, a flushing lantern (7) can be installed. By means of pressure regulation (8), for example *via* a pneumatic drive (9), adjustment is possible in fractions of a second, in order to connect through or shut off the required number of nozzles to ensure that the homogenising pressure and hence the emulsion quality are always the same. If electric step motors are used, adjustment within the ms range is also possible. Almost step-free adjustment is achieved according to Figure 2 if two opposing rows of nozzles are offset relative to each other.

It has been found to be especially advantageous if, in accordance with Figure 3, slots are used instead of the nozzles. It has been found that, if the slots are only as wide as the bore diameter, or even smaller, a very constant operation and linear, completely step-free adjustment is possible. With the apparatus according to the invention it is

possible to produce two-component polyurethane lacquers of the highest quality and with a large adjustment range.

5 The geometry of the bores and slots is to be such that energy densities of from 10^5 to 10^7 W/cm³, preferably from 10^6 to 10^7 W/cm³, are achieved. That is achieved if, in accordance with Figures 2 and 3, the amount of material removed in the region of the bore or of the slot is such that the length of the bore is from 1 to 3 times as long as the diameter of the bore or the width of the slot. Especially preferably from 1 to 2 times as long.

10 The jet disperser that has been developed additionally has the advantage that an intermediate buffer and hence the outlay in terms of flushing when the apparatus is at a standstill can be minimised, if the polyisocyanate is continuously emulsified immediately before being introduced into the spray gun or atomiser bell.

15 Figure 4 shows the embodiment according to the invention, for example, for the initial lacquering of motor vehicles. Polyol and polyisocyanate are pumped continuously and without pulsation to the mixing nozzle (2) and the adjustable jet disperser (3) *via* one or more metering pumps (1). The metering pumps also serve to
20 apply the necessary pump pressure. After production of the emulsion, the two-component lacquer can be stored *via* a small buffer (4) for a plurality of atomising bells (5), if the lacquer is to be atomised with internal electrical charging. In the case of external electrical charging, it is possible to dispense with the buffers.

25 By means of the process according to the invention it is possible to produce, for example, bimodal aqueous two-component polyurethane lacquer emulsions based on hydroxy-functional resin dispersions and polyisocyanates, which have a particle size distribution having a first distribution maximum at a particle size of <500 nm, preferably from 10 to 200 nm, and a second distribution maximum at a particle size
30 of from 200 to 2000 nm. The second distribution maximum is preferably at a particle

size of from 300 to 1000 nm. The particle sizes of the distribution maxima differ at least by a factor of 2.

5 In particular, 99 wt.% of the particles in the emulsion according to the invention have a particle size of less than 5000 nm, preferably less than 1000 nm.

According to the invention, all binders and crosslinking components hitherto used for two-component polyurethane lacquers can be employed.

10 Suitable binder resins are, for example, polyacrylates, polyesters, urethane-modified polyesters, polyethers, polycarbonates or polyurethanes containing isocyanate-reactive groups, especially those in the molecular weight range of from 1000 to 10,000 g/mol. Hydroxyl groups are preferably used as the isocyanate-reactive groups. The binder resins are generally used in the form of aqueous dispersions.

15 There are suitable as the polyisocyanate component any organic polyisocyanates having aliphatically, cycloaliphatically, araliphatically and/or aromatically bonded, free isocyanate groups. In general, the polyisocyanate component is to have a viscosity of from 20 to 1000 mPa.s, preferably of less than 500 mPa.s. However,
20 polyisocyanates having a higher viscosity may also be used if the viscosity of the polyisocyanate component is reduced by an appropriate solvent content.

It is especially preferred to use as the polyisocyanates those having exclusively aliphatically and/or cycloaliphatically bonded isocyanate groups having an average
25 NCO functionality of from 2.2 to 5.0 and a viscosity of from 50 to 500 mPa.s at 23°C. At a correspondingly lower viscosity, dispersion is possible according to the invention with a sufficiently small particle size entirely without the addition of solvent.

It is also possible to use the conventional additives and modifying agents known in lacquer chemistry.

5 The process according to the invention is not tied to the use of component systems which have been developed specifically for water-dispersible lacquer systems, as described, for example, in the literature cited at the beginning. Rather, it is possible according to the invention to use a large number of the two-component systems which hitherto have not been dispersible in water. In general, however, where two-
10 accordance with the invention, the outlay in terms of dispersion within the scope of the present invention (that is to say, the pressure to be applied) will be especially advantageous.

15 The emulsions obtained in the process according to the invention are suitable for the production of high-quality coatings on a wide variety of substrates and materials, such as wood, metals, plastics, etc.. The coating systems are used preferably for the lacquering of bodies or body parts in the initial lacquering of motor vehicles.

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Examples

Example 1

- 5 Continuous production of a paraffin oil emulsion having the following recipe:
- 4 parts of paraffin oil, highly liquid
 - 1 part of emulsifier, Tween 80/Arlacel 80 mixture, HLB 11.5, and
 - 5 parts of water.
- 10 In Figure 5, the test results obtained with an adjustable hole-type nozzle according to Figure 2 having 10 0.1 mm holes, with a slot-type nozzle having a width of 0.1 mm and a height of 6 mm, and with a jet disperser having fixed dimensions and having 2 0.1 mm bores, are compared by means of a graph.
- 15 For the adjustable nozzles, in each case the smallest openings, a middle setting and the maximum opening are recorded. The graph, which gives the average particle size as a function of the homogenising pressure, shows good correspondence over the entire range and the good functioning of the adjustable jet disperser.

20 **Example 2**

Continuous preparation of a two-component polyurethane lacquer according to the following recipe:

| | |
|--|----------|
| Binder component: | |
| Bayhydrol® VP LS 2271 (hydroxy-functional polyacrylate dispersion, Bayer AG) | 30.39 % |
| Bayhydrol® VP LS 2231 (hydroxy-functional, urethane-modified polyester dispersion, Bayer AG) | 33.28 % |
| Byk® 345 lacquer auxiliary, Byk Chemie GmbH | 0.29 % |
| Byk® 333 lacquer auxiliary, Byk Chemie GmbH | 0.30 % |
| distilled water | 7.65 % |
| Hardener component: | |
| Desmodur® VP LS 2025/1 (lacquer polyisocyanate, Bayer AG) | 18.29 % |
| Tinuvin® 1130, 50 % in butyl diglycol acetate (light stabiliser, Ciba Spezialitätenchemie GmbH) | 1.85 % |
| Tinuvin® 292, 50 % in butyl diglycol acetate (HALS stabiliser, Ciba Spezialitätenchemie GmbH) | 0.92 % |
| butyl diglycol acetate / Solvesso 100 (1/1) | 7.03 % |
| | 100.00 % |

5 The two components are mixed in accordance with Figure 4 using an adjustable jet disperser according to Figure 1 having 0.2 mm hole-type nozzles according to Figure 2, and the hardener component is emulsified.

The lacquer is applied electrostatically to galvanised steel sheets in a layer thickness of 40 µm by means of a commercially available bell. The lacquer film is exposed to

air for 5 minutes at room temperature, pre-dried for 10 minutes at 80°C and hardened for 30 minutes at 180°C. The lacquer film has the following performance properties:

| | |
|---|-----------|
| pendulum hardness according to König (23°C) | 190 s |
| 20° shine | 88 |
| resistance to solvents xylene/motor fuel (0 = very good, 5 = poor) | 0 / 1 |
| resistance to chemicals pancreatin/sulfuric acid/sodium hydroxide solution | 2 / 1 / 0 |
| scratch resistance (Amtec Kistler laboratory washing line, 10 cycles), Δ shine | 13 |

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Patent claims

- 5 1. Process for the production of aqueous two-component polyurethane lacquer emulsions, with variable throughput, based on aqueous binder dispersions containing isocyanate-reactive groups and on polyisocyanates, characterised in that the mixture is pumped under a pressure of from 1 to 30 MPa through an adjustable jet disperser having nozzle bores or slots which can be connected through or shut off.

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Fig.1

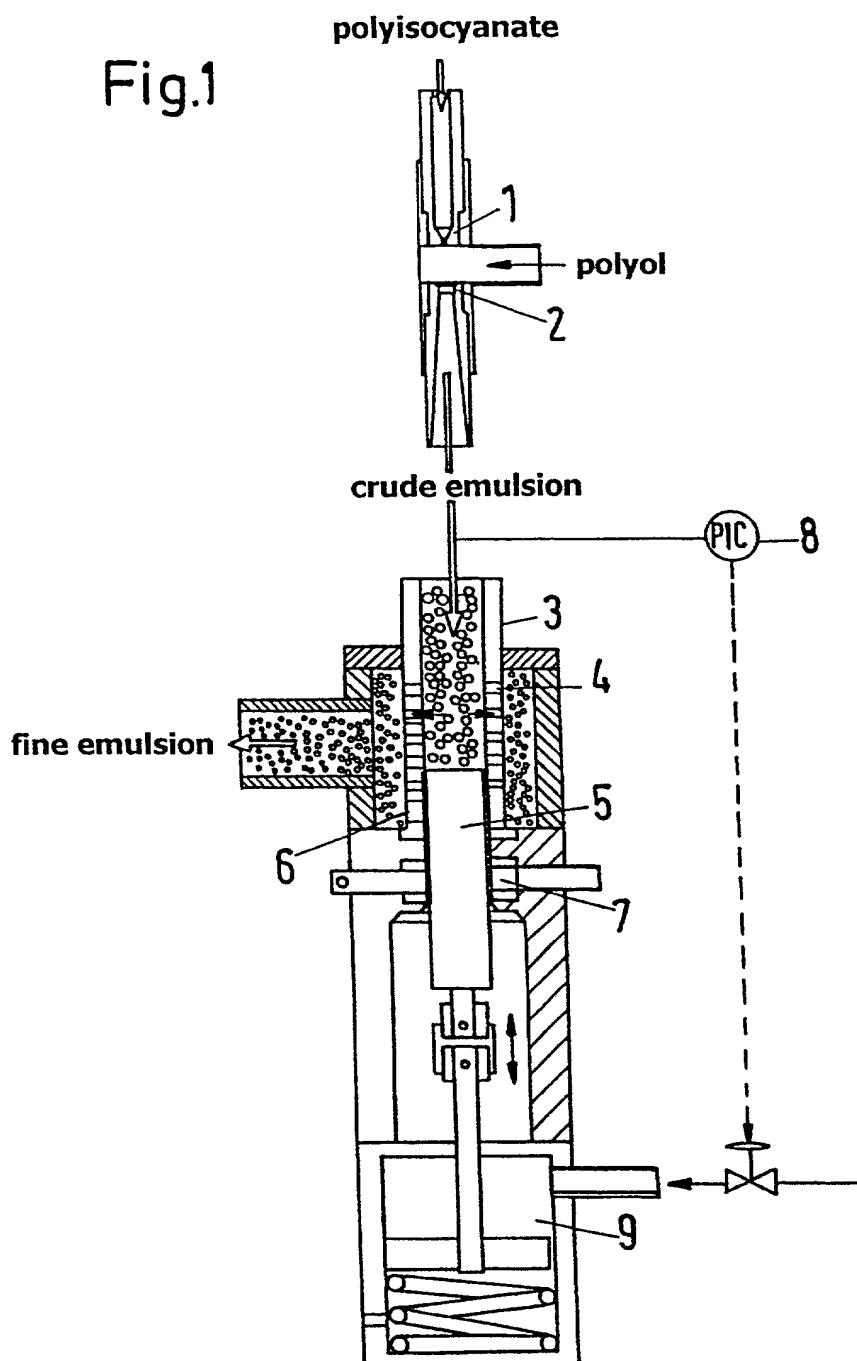


Fig.2

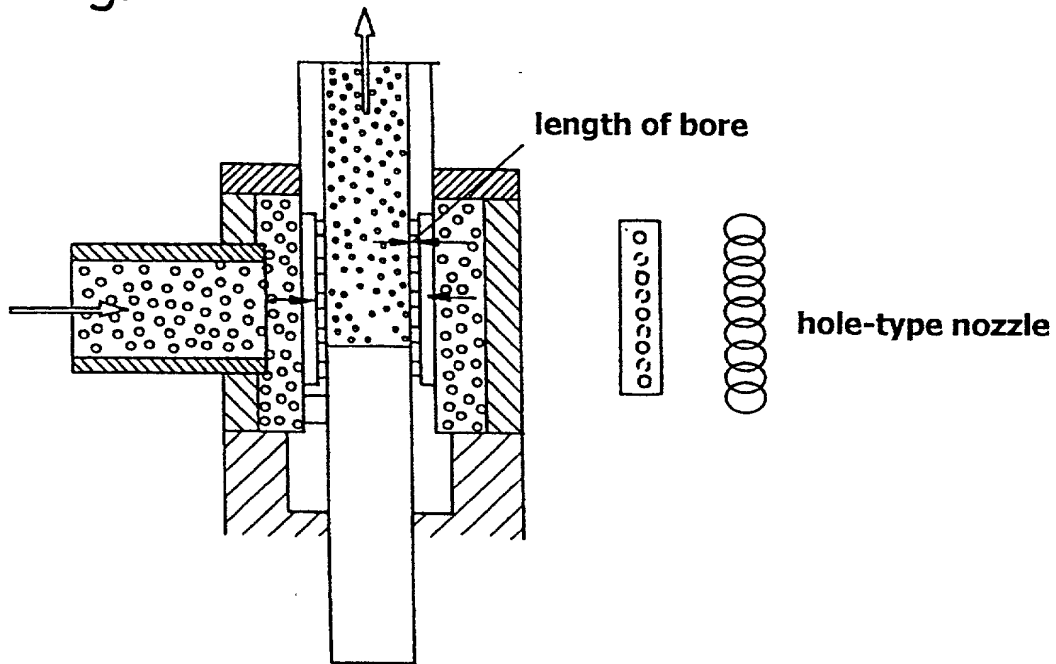
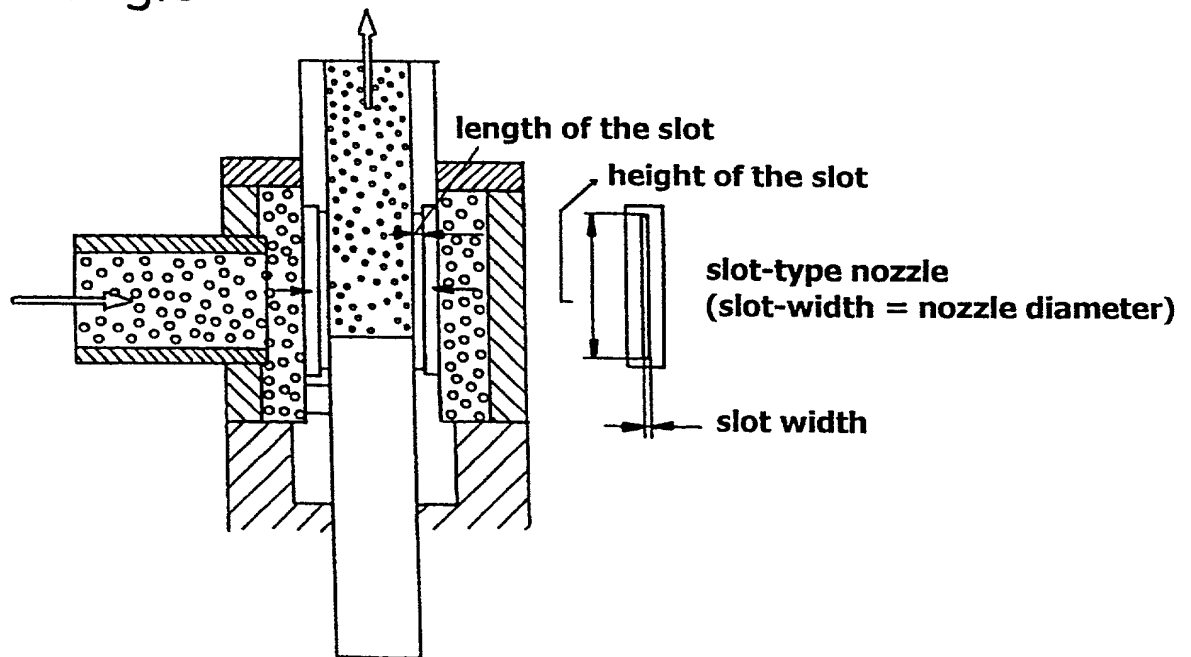


Fig.3



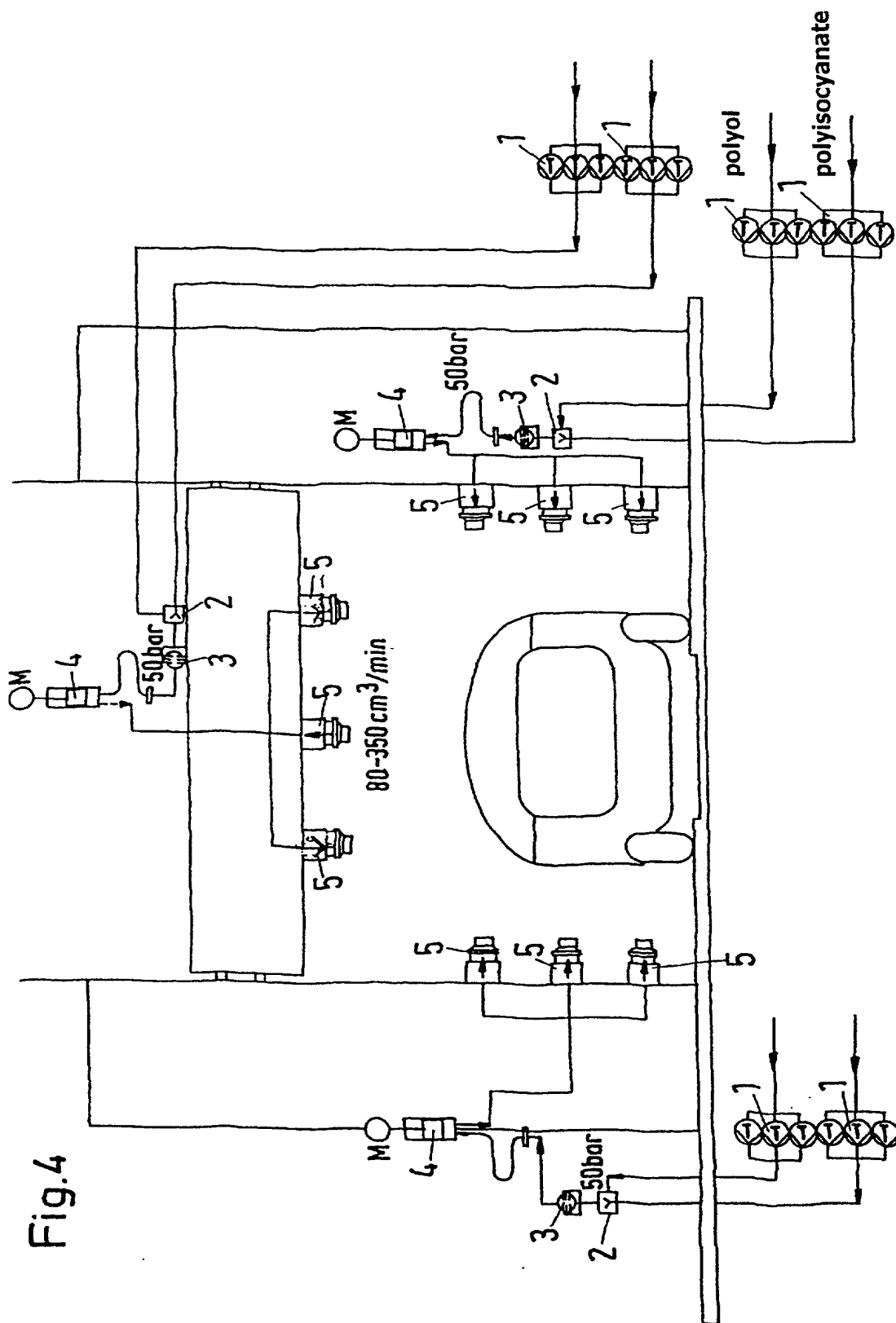
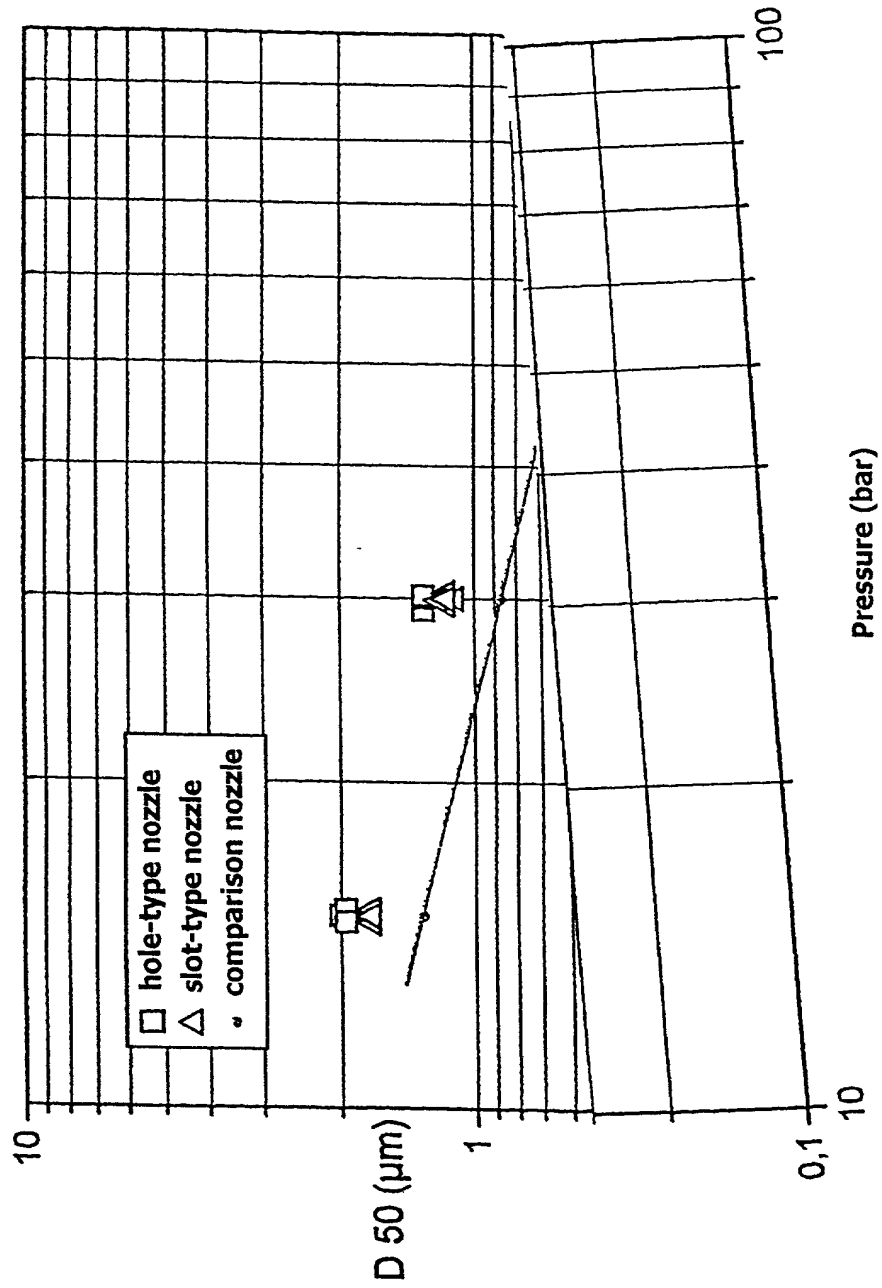


Fig. 5

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought

on the invention entitled

ADJUSTABLE JET DISPERSER FOR PRODUCING AQUEOUS TWO-COMPONENT POLYURETHANE PAINT EMULSIONS

the specification of which is attached hereto,

or was filed on **July 12, 2000**

as a PCT Application Serial No. **PCT/EP00/06599**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

199 33 441.2
(Number)

Germany
(Country)

July 16, 1999
(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Le A 33 410-US

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